(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) Publication numb r: **0 428 221 A1**

(12) **EUROPEAN PATENT APPLICATION**

(21) Application number: **90202981.8**

(22) Date of filing: **12.11.90**

(51) Int. Cl.5: **C08K 5/00**, C08L 75/04, //(C08K5/00,5:06,5:523)

(30) Priority: **14.11.89 US 436989**

(43) Date of publication of application: **22.05.91 Bulletin 91/21**

(84) Designated Contracting States: **DE FR GB NL**

(71) Applicant: **AKZO N.V.**
Velperweg 76
NL-6824 BM Arnhem(NL)

(72) Inventor: **Fesman, Gerald**
319 Vandelinda Avenue
Teaneck, New Jersey 07666(US)
Inventor: **Aaronson, Alan Michael**
65-48 173 Street
Flushing Meadows, New York 11365(US)

(74) Representative: **Schalkwijk, Pieter Cornelis et al**
AKZO N.V., Patent Department (Dept. CO),
P.O. Box 9300
NL-6800 SB Arnhem(NL)

(54) Viscosity reduction of high viscosity fluid flame retardants for polyurethanes.

(57) Triphenyl phosphate can be used to reduce the viscosity of fluid flame retardants (polybrominated aryl oxides, oligomeric phosphate esters, etc.) which are useful in flame retarding polyurethane compositions.

EP 0 428 221 A1

VISCOSITY REDUCTION OF HIGH VISCOSITY FLUID FLAME RETARDANTS FOR POLYURETHANES

BACKGROUND OF THE INVENTION

Polyurethane foams, coatings and elastomers are commonly admixed with flame retardants to achieve the desired degree of flame retardancy for the final material desired. Pumpable fluid (or liquid) flame retardant compositions are a preferred class since such pumpable formulations are needed in environments where automation and machine mixing of the various components are used. Examples of the chemical classes for such fluid or liquid flame retardants include brominated aryl flame retardants, such as polybromodiphenyl oxide, and various viscous organophosphorus flame retardants such as the oligomeric phosphate esters, such as the chlorinated oligomeric phosphate esters (e.g. FYROL 99 brand) and the reaction product of 2-chloro-1-propanol phosphate (3:1) (FYROL PCF brand) with ethylene oxide and phosphorus pentoxide and tris(haloalkyl) phosphates such as tris(dichloropropyl) phosphate.

High viscosity in such fluid or liquid flame retardant compositions can be disadvantageous since it makes the pumping and movement of such compositions more difficult thereby complicating manufacturing operations. For example, it is known that polybrominated diphenyl oxide flame retardants are very viscous. U.S. Patent No. 4,746,682 to J. Green indicates that either alkyl diphenyl phosphates or alkylated triaryl phosphates can be used to achieve blends having acceptably low viscosity as compared to the polybrominated aryl flame retardant itself.

DESCRIPTION OF THE INVENTION

Surprisingly, it has now been found that triphenyl phosphate, a solid at room temperature, is an effective viscosity reducing agent in the aforementioned types of liquid, but viscous, flame retardant compositions.

The level of use of triphenyl phosphate with a particular flame retardant will depend upon its initial compatibility or miscibility with the flame retardant component whose viscosity is initially high and in need of reduction. The level of triphenyl phosphate to use is dependent upon the ultimate viscosity that is desired for the composition containing it. The level of triphenyl phosphate needed to achieve such a viscosity result is, in turn, dependent upon the miscibility characteristics between the flame retardant chosen and the triphenyl phosphate. Generally, the amount of triphenyl phosphate that needs to be added will range from about 5% to about 25%, by weight of the fluid or liquid flame retardant whose viscosity is to be reduced.

The liquid flame retardants to which the instant invention can be added include those containing a major amount of a polybrominated diphenyl oxide and a minor amount of an aromatic bisphosphate (e.g., resorcinol bis(diphenyl phosphate) or the bis(diphenyl phosphate ester) of neopentyl glycol) as shown in Examples 1-6 below. Generally speaking, it has been found that up to about 25%, by weight of triphenyl phosphate, based on the weight of the entire composition, can be used in such systems.

The triphenyl phosphate viscosity reducing additive of the instant invention can also be used in similar amount with flame retardant compositions which contain oligomeric phosphate esters as the sole or predominant component. This is shown in Examples 10-12, below.

The instant invention allows for the obtaining of low enough viscosities to achieve pumpable flame retardant compositions which are required to satisfactorily process flexible and rigid polyurethane foams, for example. Easier machine mixing and miscibility are achieved by bringing the viscosities of the components closer and preferably lower. Coatings and elastomers also require low viscosities for better flowability and processing.

EXAMPLES 1-3

Th following illustrat th viscosity characteristics (cps at 25° C) of various blends containing pentabromodiphenyl oxide (PBDPO) and resorcinol bis(diphenyl phosphate) flame retardant with (Examples 2-3) and without (Example 1) triphenyl phosphate (TPP). The TPP-containing blend was heated slightly above the melting point of the TPP to achieve its dissolution:

	Components	Wt %	Viscosity
1.	PBDPO	70	
	Bisphosphate	30	9000
2.	PBDPO	70	
	Bisphosphate	20	
	TPP	10	2500
3.	PBDPO	70	
	Bisphosphate	10	
	TPP	20	900

EXAMPLES 4-6

These Examples are analogous to those in Examples 1-3 with the bisphosphate being the bis(diphenyl phosphate ester) of neopentyl glycol as more particularly described in U.S. Ser. No. 374,716, filed July 3, 1989:

	Components	Wt %	Viscosity
4.	PBDPO	70	
	Bisphosphate	30	4000
5.	PBDPO	70	
	Bisphosphate	20	
	TPP	10	2040
6.	PBDPO	70	
	Bisphosphate	10	
	TPP	20	800

EXAMPLES 7-9

These Examples are analogous to the foregoing with the flame retardant being (isopropylated phenyl diphenyl phosphate PHOSFLEX 41P brand from Akzo Chemicals Inc.):

	Components	Wt %	Viscosity
7.	PBDPO	70	
	PHOSFLEX 41P brand	30	2500
8.	PBDPO	70	
	PHOSFLEX 41P brand	20	
	TPP	10	950
9.	PBDPO	70	
	PHOSFLEX 41P brand	10	
	TPP	20	625

EXAMPLES 10-12

In these Examples the pentabromodiphenyl oxide of Examples 1-9 is replaced by the reaction product (3:1) of 2-propanol-1-chlorophosphate with ethylene oxide and phosphoric oxide [CAS Registry No. 72102-42-2), referred to below as "RP":

	Components	Wt %	Viscosity
10.	RP	100	7700
11.	RP	90	
	PHOSFLEX 41P brand	10	4900
12.	RP	90	
	TPP	10	3000

EXAMPLES 13-15

Tris(dichloropropyl) phosphate (FYROL FR-2 brand from Akzo Chemicals Inc.), a successful commercial flame retardant, is even more desirable if its viscosity of 2000 cps at 25°C could be reduced to around 1000 cps with a minimum use level of a compatible additive which would not severely reduce its flame retardancy effectiveness. The following illustrates the desired effect, in accordance with the present invention, in Example 15:

	Components	Wt %	Viscosity
13.	FYROL FR-2 brand	100	1200
14.	FYROL FR-2 brand	95	
	PHOSFLEX 41P brand	5	1200
15.	FYROL FR-2 BRAND	95	
	TPP	5	950

Claims

1. A fluid flame retardant composition suitable for use in forming flame retarded polyurethane composition which comprises a normally viscous flame retardant and an effective amount of triphenyl phosphate for viscosity reduction thereof.

2. A composition as claimed in Claim 1 wherein the flame retardant composition comprises a major amount of a polybrominated diphenyl oxide.

3. A composition as claimed in Claim 2 wherein the flame retardant is pentabromodiphenyl oxide.

4. A composition as claimed in Claim 1 wherein the flame retardant composition comprises a major amount of an oligomeric phosphate ester.

5. A composition as claimed in any one of the preceding Claims wherein the triphenyl phosphate is present at up to about 25%, by weight of the entire composition.

European Patent Office

EUROPEAN SEARCH REPORT

Application Number

EP 90 20 2981

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	DE-A-3 005 477 (CHEMISCHE FABRIK KALK GMBH) * page 4, lines 8 - 25; claims *	1-5	C 08 K 5/00 C 08 L 75/04 // (C 08 K 5/00 C 08 K C 08 K 5:06 C 08 K 5:523)
D,A	US-A-4 746 682 (JOSEPH GREEN) * column 2, line 61 - column 3, line 66; claims *	1-5	
A	WO-A-8 903 403 (GREAT LAKES CHEMICAL CORPORATION) * page 6, lines 12 - 22; claims *	1-5	
A	FR-A-2 304 642 (CHEMISCHE FABRIK KALK GMBH) * example 8; claims *	1-5	

TECHNICAL FIELDS SEARCHED (Int. Cl.5)

C 08 K
C 08 L

The present search report has been drawn up for all claims

Place of search	Date of completion of search	Examiner
Th Hague	13 February 91	DE LOS ARCOS Y VELAZ

CATEGORY OF CITED DOCUMENTS

X: particularly relevant if taken alone
Y: particularly relevant if combined with another document of the same catagory
A: technological background
O: non-written disclosure
P: intermediate document
T: theory or principle underlying the invention

E: earlier patent document, but published on, or after the filing date
D: document cited in the application
L: document cited for other reasons

&: member of the same patent family, corresponding document